

METHOD 7550

OSMIUM (ATOMIC ABSORPTION, DIRECT ASPIRATION)

1.0 SCOPE AND APPLICATION

1.1 Method 7550 is an atomic absorption procedure approved for determining the concentration of osmium in wastes, mobility procedure extracts, soils, and ground water. All samples must be subjected to an appropriate dissolution step prior to analysis.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis by Method 7550, samples must be prepared for direct aspiration. The method of sample preparation will vary according to the sample matrix. Aqueous samples are subjected to the acid digestion procedure discussed in this method. Sludge samples are prepared using the procedure described in Method 3050. For samples containing oils, greases, or waxes, the procedure described in Method 3040 may be applicable. Due to the very volatile nature of some osmium compounds, the applicability of a method to a sample must be verified by means of spiked samples or standard reference materials, or both.

2.2 Following the appropriate dissolution of the sample, a representative aliquot is aspirated into a nitrous oxide/acetylene flame. The resulting absorption of hollow cathode radiation will be proportional to the osmium concentration. Background correction must be employed for all analyses.

2.3 The typical detection limit for this method is 0.3 mg/L; typical sensitivity is 1 mg/L.

3.0 INTERFERENCES

3.1 Background correction is required because nonspecific absorption and light scattering can be significant at the analytical wavelength.

3.2 Due to the volatility of osmium, standards must be made on a daily basis, and the applicability of sample-preparation techniques must be verified for the sample matrices of interest.

3.3 Samples and standards should be monitored for viscosity differences that may alter the aspiration rate.

3.4 Osmium and its compounds are extremely toxic; therefore, extreme care must be taken to ensure that samples and standards are handled properly and that all exhaust gases are properly vented.

4.0 APPARATUS AND MATERIALS

4.1 Atomic absorption spectrophotometer: Single- or dual-channel, single- or double-beam instrument with a grating monochromator, photomultiplier detector, adjustable slits, and provisions for background correction.

4.2 Osmium hollow cathode lamp.

4.3 Strip-chart recorder (optional).

5.0 REAGENTS

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Concentrated nitric acid (HNO₃): Acid should be analyzed to determine levels of impurities. If a method blank using the acid is <MDL, the acid can be used.

5.3 Osmium standard stock solution (1,000 mg/L): Procure a certified aqueous standard from a supplier and verify by comparison with a second standard. If necessary, standards can be made from osmium compounds. However, due to the toxicity of these compounds, this approach is not advised.

5.4 Osmium working standards: Prepare dilution of the stock solution at the time of analysis. These standards should be prepared to contain 1% (v/v) HNO₃ and 1% (v/v) H₂SO₄.

5.5 Air: Cleaned and dried through a suitable filter to remove oil, water, and other foreign substances. The source may be a compressor or a cylinder of industrial-grade compressed air.

5.6 Acetylene: Should be of high purity. Acetone, which is usually present in acetylene cylinders, can be prevented from entering and affecting flame conditions by replacing the cylinder before the pressure has fallen to 50 psig.

5.7 Nitrous oxide: Cylinder suitable for instrumental analysis.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Chapter Nine of this manual.

6.2 All sample containers must be prewashed with detergents, acids, and Type II water. Plastic and glass containers are both suitable.

6.3 Aqueous samples must be acidified to a pH <2 with HNO₃.

6.4 Nonaqueous samples shall be refrigerated, when possible, and analyzed as soon as possible.

7.0 PROCEDURE

7.1 Sample preparation: Aqueous samples should be prepared according to the procedure described in Paragraph 7.2. Sludge-type samples should be prepared according to Method 3050; samples containing oils, greases, or waxes may be prepared according to Method 3040. The applicability of a sample preparation technique to a new matrix type must be demonstrated by analyzing spiked samples, relevant standard reference materials, or both.

7.2 Sample preparation of aqueous samples:

7.2.1 Transfer a representative 100-mL aliquot of the well-mixed sample to a Griffin beaker and add 1 mL of concentrated HNO_3 .

7.2.2 Place the beaker on a steam bath or hot plate and warm for 15 min. Cool the beaker and, if necessary, filter or centrifuge to remove insoluble material.

7.2.3 Add 1 mL of concentrated H_2SO_4 and adjust the volume back to 100 mL. The sample is now ready for analysis.

7.3 The 290.0-nm wavelength line and background correction shall be employed.

7.4 A fuel-rich nitrous oxide/acetylene flame shall be used.

7.5 Follow the manufacturer's operating instructions for all other instrument parameters.

7.6 Either (1) run a series of osmium standards and construct a calibration curve by plotting the concentrations of the standards against the absorbances, or (2) for the method of standard additions, plot added concentration versus absorbance. For instruments that read directly in concentration, set the curve corrector to read out the proper concentration.

7.7 Analyze all EP extracts, all samples analyzed as part of a delisting petition, and all samples that suffer from matrix interferences by the method of standard additions.

7.8 Duplicates, spiked samples, and check standards should be routinely analyzed.

7.9 Calculate metal concentrations: (1) by the method of standard additions, (2) from a calibration curve, or (3) directly from the instrument's concentration read-out. All dilution or concentration factors must be taken into account.

8.0 QUALITY CONTROL

8.1 All quality control data should be maintained and available for easy reference or inspection.

8.2 Calibration curves must be composed of a minimum of a blank and three standards. A calibration curve should be made for every hour of continuous sample analysis.

8.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

8.4 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.5 Verify calibration with an independently prepared check standard every 15 samples.

8.6 Run one spike duplicate sample for every 10 samples. A duplicate sample is a sample brought through the entire sample preparation and analytical process.

8.7 The method of standard additions (see Method 7000, Section 8.7) shall be used for the analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

9.0 METHOD PERFORMANCE

9.1 Precision and accuracy data are not available at this time.

10.0 REFERENCES

1. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-82-055, December 1982, Method 252.1.

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